132nd Annual TMS Meeting - March 2nd-6th, 2003

Deformation and Failure in Dentin:
A Mechanistic and Fracture Mechanics Based Approach

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with

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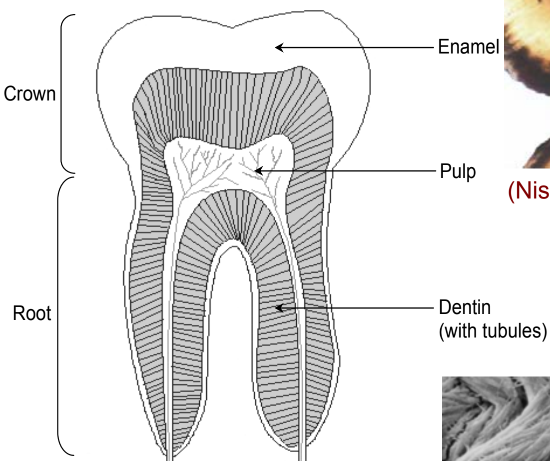
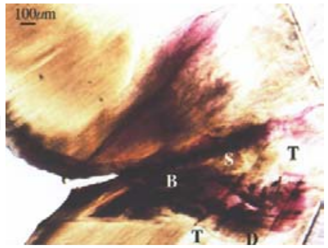
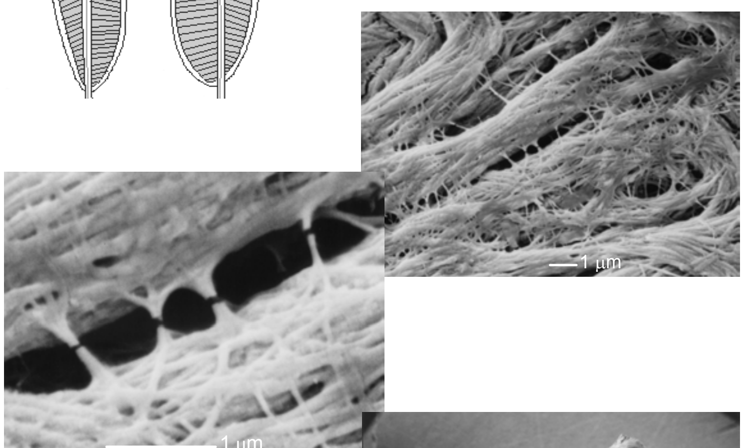
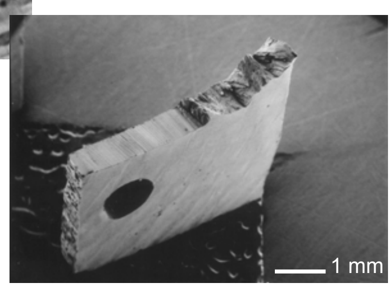
Motivation and Significance





(Nishikawa et *al*., 2003)





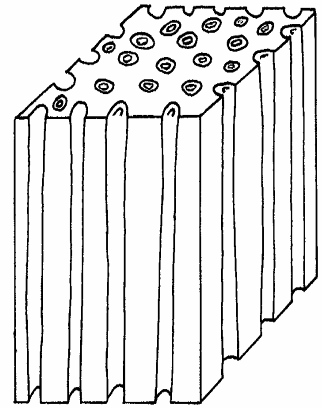
- Dentin is the most abundant mineralized tissue in teeth, and is critical for structural integrity
- Fracture and fatigue properties of dentin are an issue of obvious clinical relevance
- Non-carious notches in exposed root surfaces are sites for fracture and fatigue cracking
- Very few *fracture mechanics* based studies reported in archival literature
- Mechanical properties of dentin have implications for other mineralized tissues, such as bone

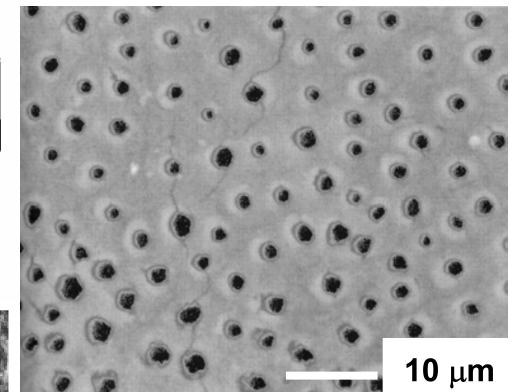
Objectives

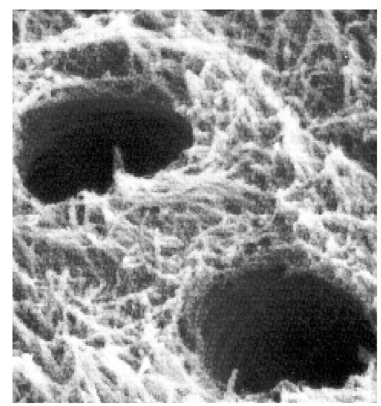
- Measure the mechanical properties, specifically fracture toughness, fatigue and subcritical crack-growth properties, of dentin, as a function of orientation
- Characterize the micro-mechanisms of fracture in terms of the underlying features, and anisotropy, of the microstructure
- Identify and model the salient toughening mechanisms in dentin
- Establish a mechanism for the fatigue of dentin
- Develop a physical basis for a damage-tolerant life-prediction methodology for teeth

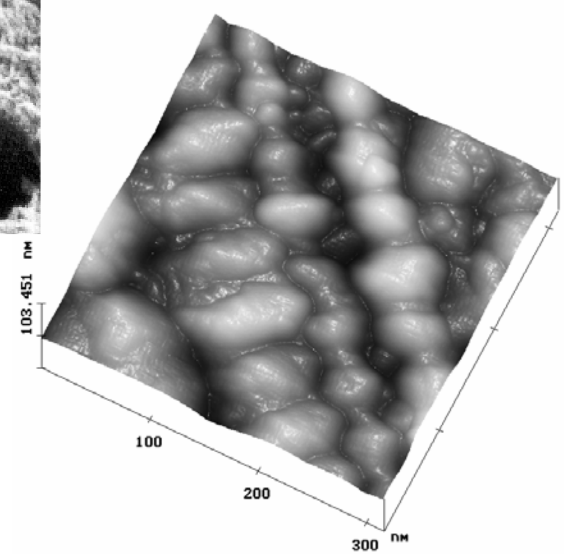
Microstructure of Dentin

- Distinctive feature are 1-2 μm dia. cylindrical tubules, running from the dentin-enamel junction to the soft, interior pulp
- Hydrated composite of nanocrystalline carbonated apatite mineral (~45% vol.), collagen fibrils (~30% vol.) and fluid (~25% vol.)
- Mineral crystallites (5 nm thick) distributed in a scaffold of collagen fibers (50-100 nm dia.)
- Collagen fibrils form a planar felt-like structure oriented perpendicular to the tubules
- A simplified model for other mineralized tissue, such as bone?









Kinney, Marshall & Marshall, *Crit Rev Oral Biol*, in press (2003)

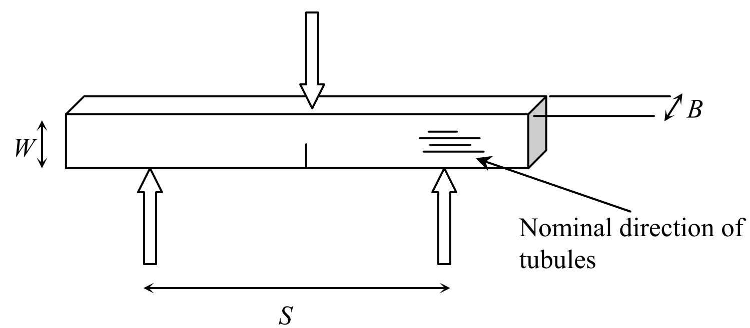
Fracture and Fatigue of Dentin: Objectives

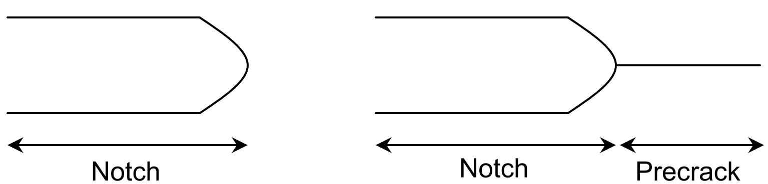
- What is the *macroscopic* fracture toughness of dentin?
- What is the *local* criterion for fracture in dentin?
- Does the highly directional nature of the microstructure affect the toughness and crack growth?
- Mechanistically, what are the origins of toughness in dentin?
  - What effect do the tubules have, either by blunting or deflecting cracks?
  - Can the collagen fibrils promote crack bridging?
  - Are there other salient toughening mechanisms?
- What is the nature of inelasticity (“yielding”) in dentin?
- What is the nature of fatigue and cyclic damage in dentin?

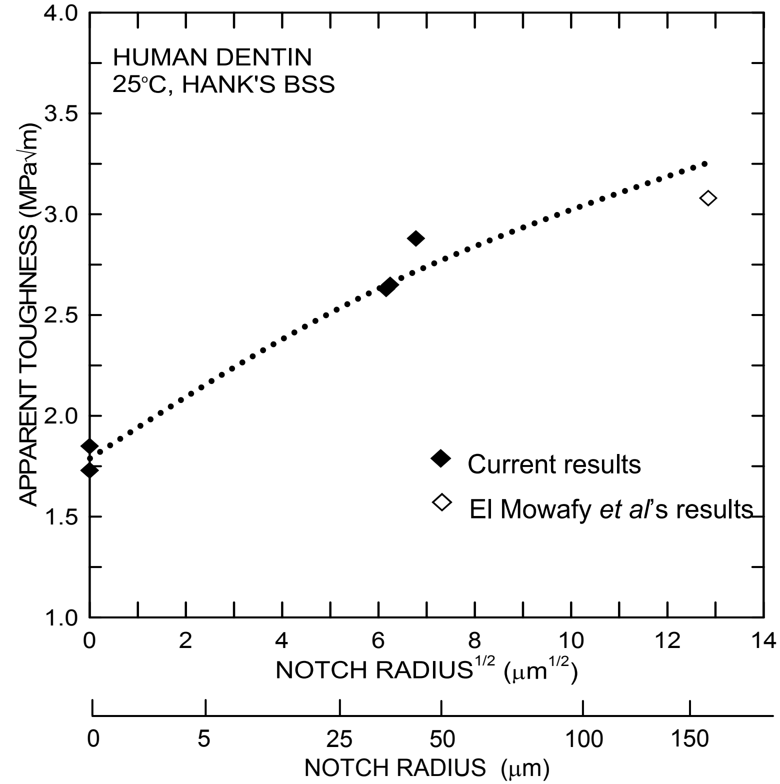
Fracture Toughness of Dentin

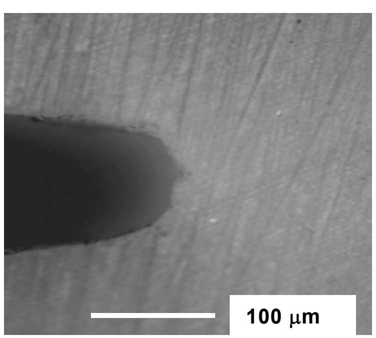
- Rasmussen *et al.*, (1976 & 1984)
  - first study of the toughness of dentin approach
  - found an orientation dependence on toughness – material was tougher parallel to tubules than perpendicular to tubules
  - toughness was measured by "work of fracture"; results are size and geometry dependent
- el Mowafy *et al.*, (1986)
  - first fracture-mechanics based study - using C(T) samples
  - measured K_c = 3.08 MPa√m for fracture parallel to tubules
  - overestimate of toughness as notched, not precracked, sample used
- Ruse *et al.*, (2001)
  - used so-called "Notchless Triangular Prism" technique
  - reported orientation-dependent values of K_c = 1.13 - 2.02 MPa√m
  - non-standard test configuration

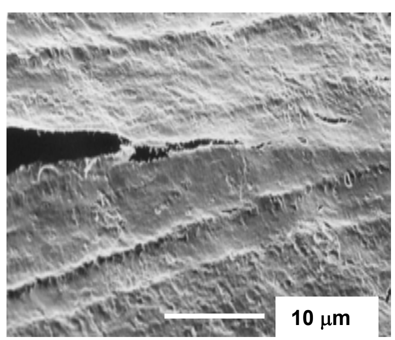
Fracture Toughness – Notch vs. Precrack









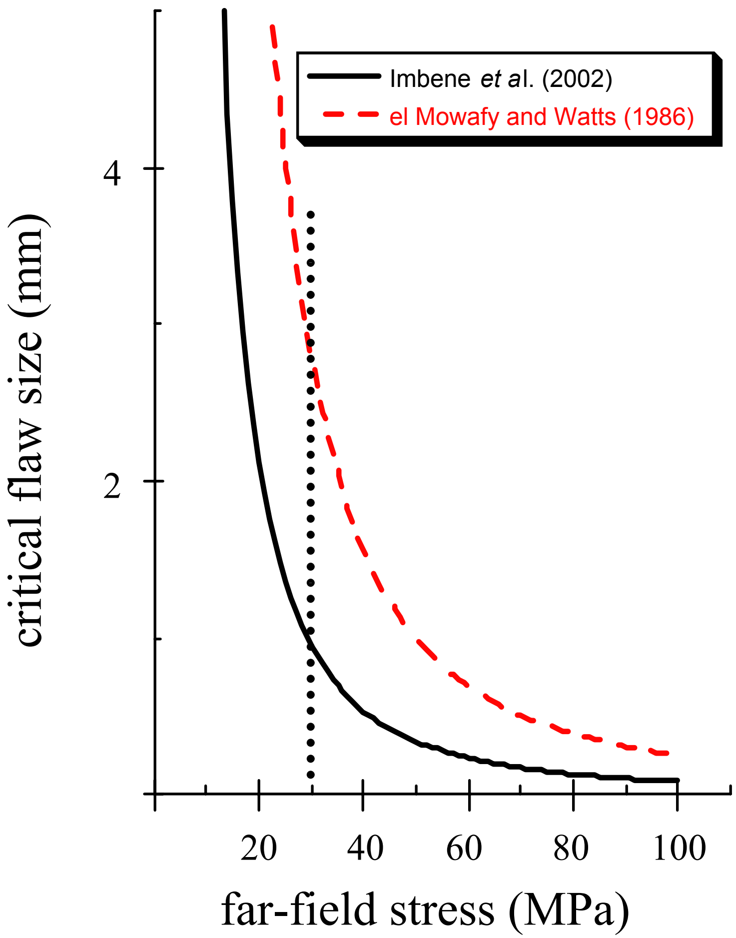


Definite effect of precrack acuity on K_c:

- Notch toughness = 2.7 MPa√m (s.d. 0.1)
  320 J/m^{-2} (s.d. 0.4)
- Precrack toughness = 1.8 MPa√m (s.d. 0.1)
  140 J/m^{-2} (s.d. 0.4)

Imbeni, Nalla, Bosi, Kinney & Ritchie, *J Biomed Mater Res*, in press (2003)

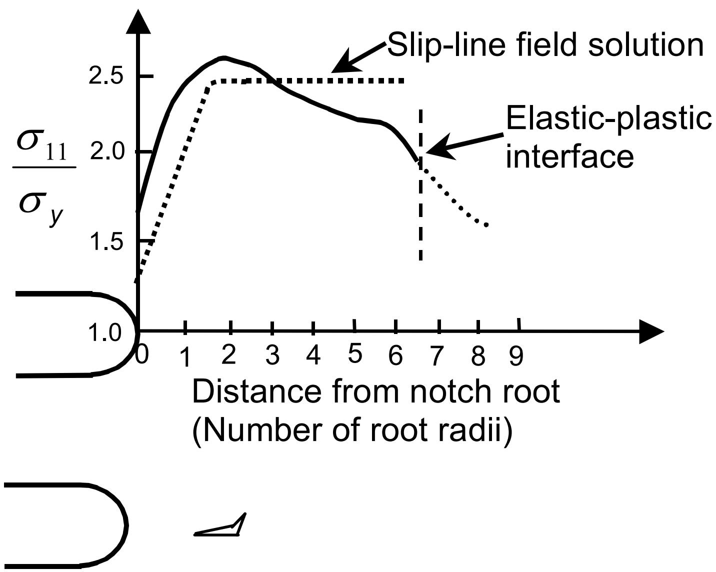
Estimation of Critical Flaw Sizes

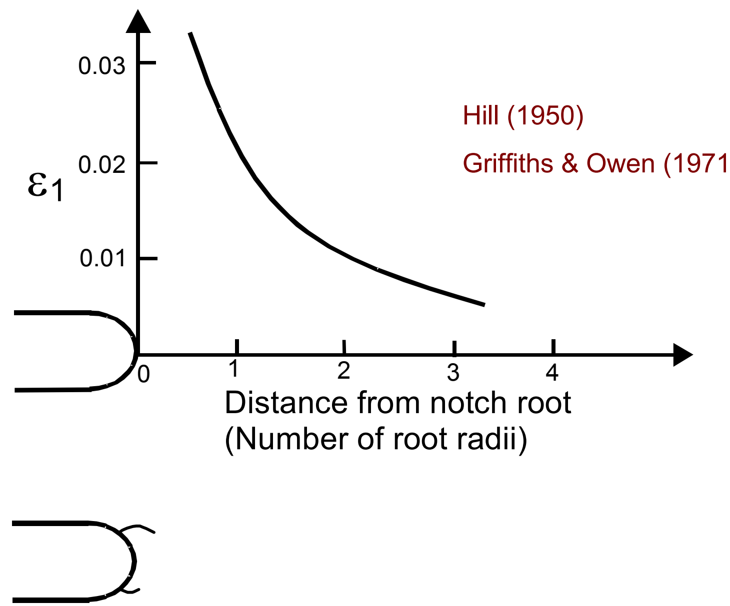


- Lower estimate of $K_c < 2$ MPa√m significantly reduces the critical flaw size in dentin
- For comparison:
  - dental cements $K_c \sim$ 0.1-0.5 MPa√m
  - amalgams $K_c \sim$ 0.1-1.6 MPa√m
  - dental composites $K_c \sim$ 0.6-2.0 MPa√m
- However, catastrophic fracture is not the only problem - subcritical crack growth, e.g., by fatigue, is also an issue
- Life prediction based on time (or cycles) for flaws to grow subcritically

Stress- *vs.* Strain-Controlled Fracture

- Fracture of mineralized tissue invariably modeled as *strain-controlled*
- However, there is no experimental support for this hypothesis



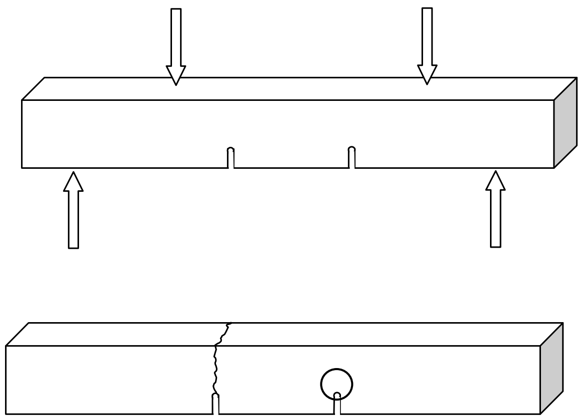


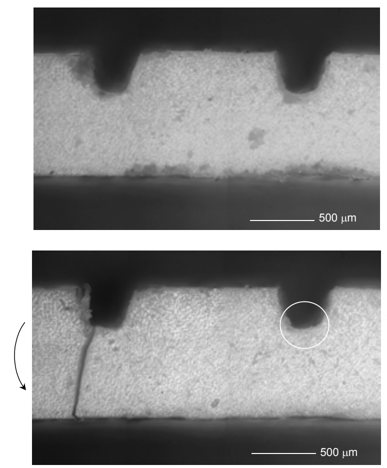
For fracture at a notch in a material displaying some degree of inelasticity

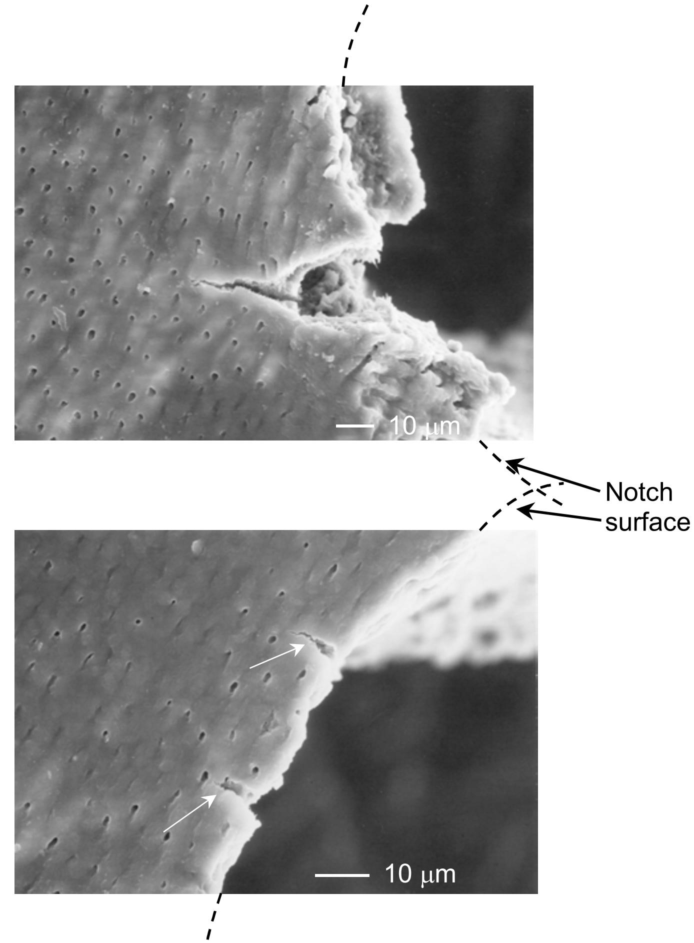
- **stress-controlled fracture:** initiates *ahead* of the notch
- **strain-controlled fracture:** initiates *at* the notch

Double-Notch Four-Point Bend Test

- Two identical notches in a four-point bend bar
- Constant bending moment on both notches
- One notch breaks - the other *freezes* local microstructural events just prior to fracture
- A good way to obtain stable cracks in dentin





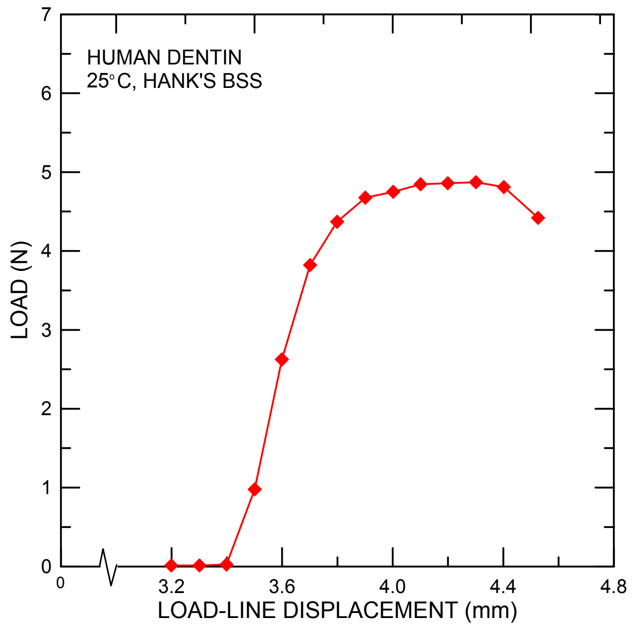


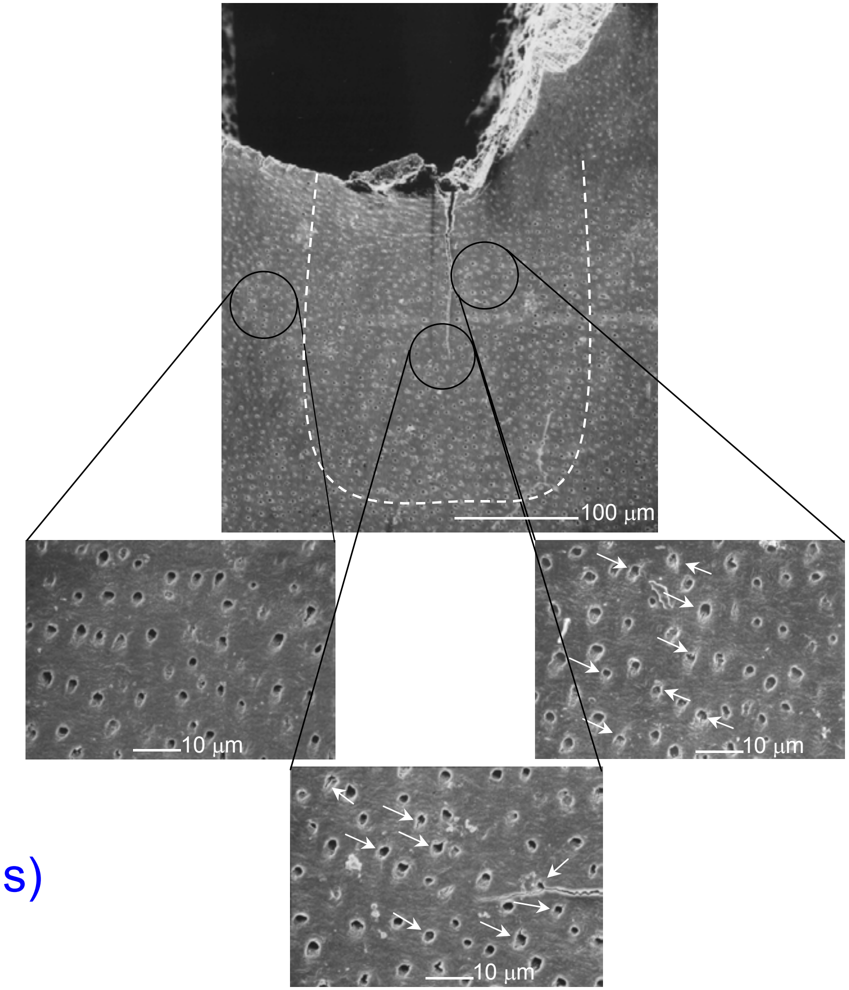
- Crack initiation directly *at* the notch root provides definite evidence that fracture in dentin is ***strain-controlled***

Nalla, Kinney & Ritchie, *J Biomed Mater Res*, in press (2003)

Nature of Inelasticity in Dentin

- **uniaxial tensile test**





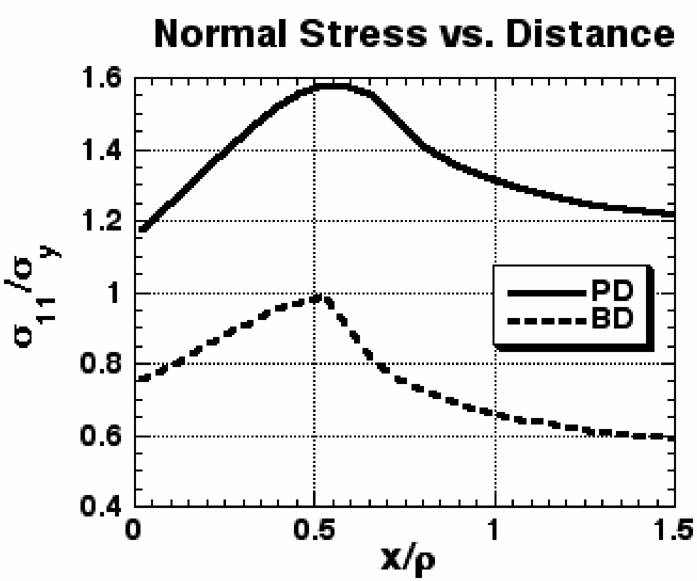
Inelastic deformation results from:

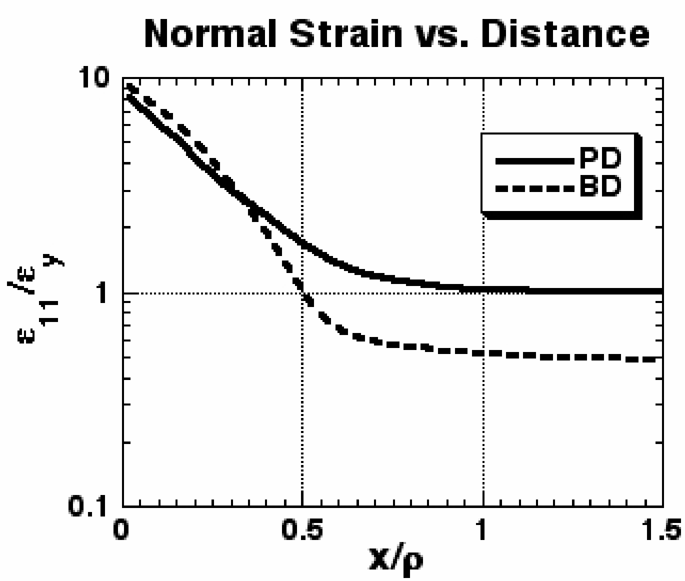
- plastic deformation (in the collagen fibrils)
- microdamage (at the peritubular cuffs)
- poro-elasticity (from fluid in the tubules)

Nalla, Kinney & Ritchie, *J Biomed Mater Res*, in press (2003)

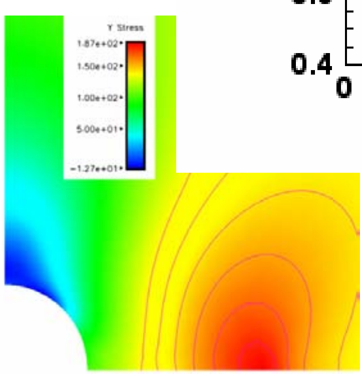
Plastic Damage *vs.* Brittle Damage

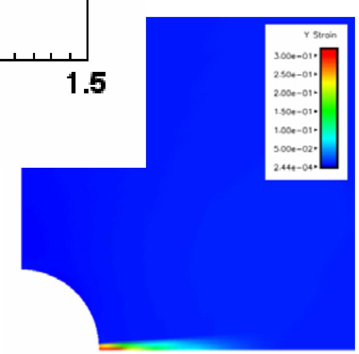
- Finite element simulation, using NIKE3D, of deformation by plasticity, using *Plastic Damage (PD) model* (Niebur *et al.*, 2000), and microcracking, using *Brittle Damage (BD) model* (Govindjee *et al.*, 1995)
- For both pressure-insensitive plasitcity and pressure-sensitive microcracking, notch-field stress and strain distributions are *qualitatively* similar





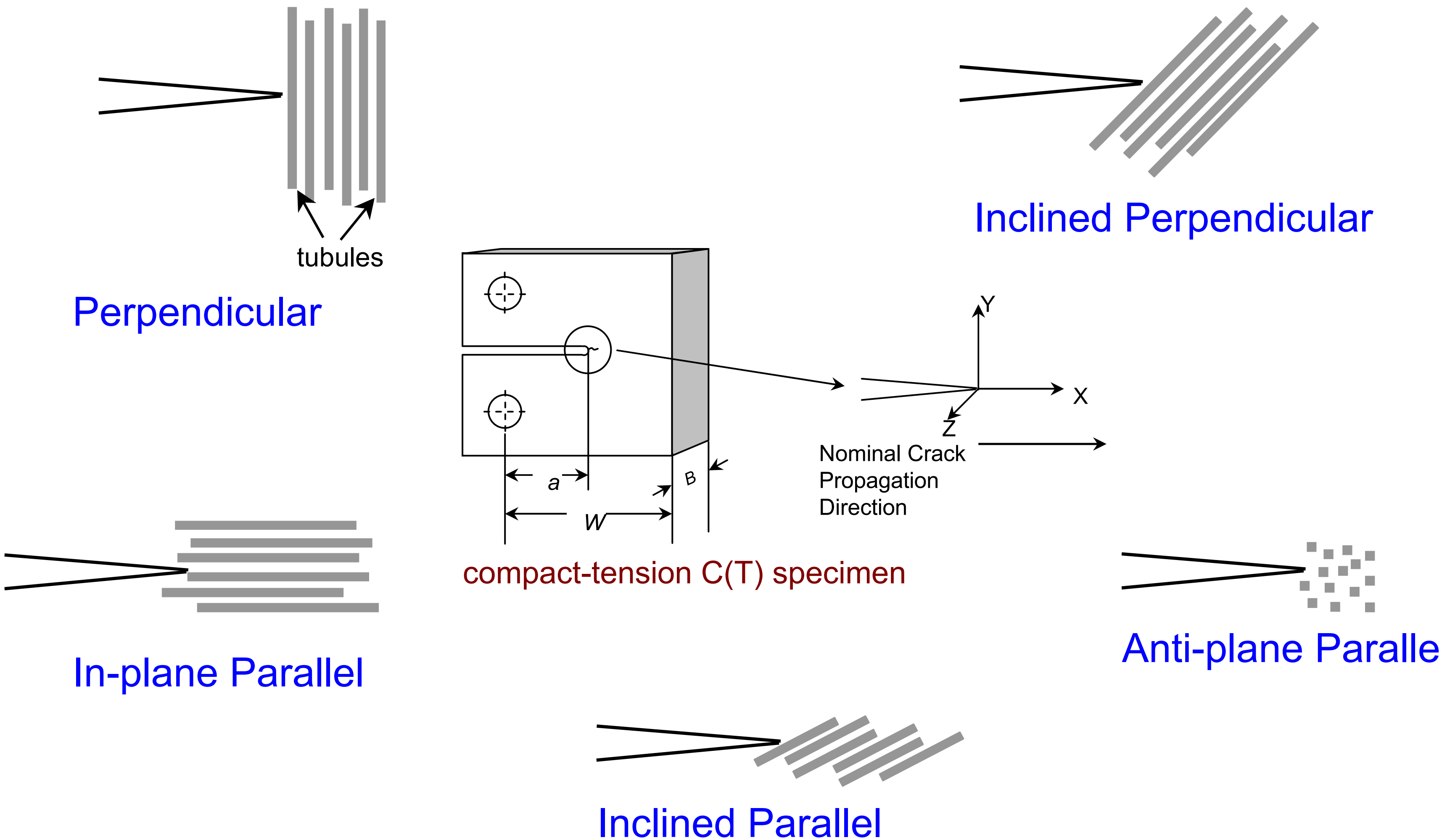
Distance ahead of notch, normalized by the notch-root radius





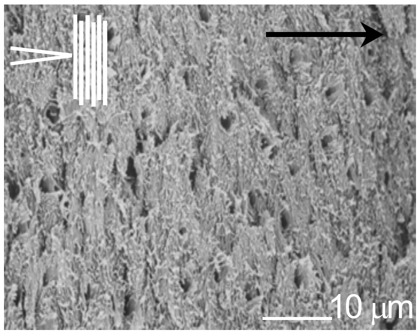
Nalla, Stölken, Kinney & Ritchie, *J Biomech*, in review (2003)

Toughness of Dentin – Effect of Orientation



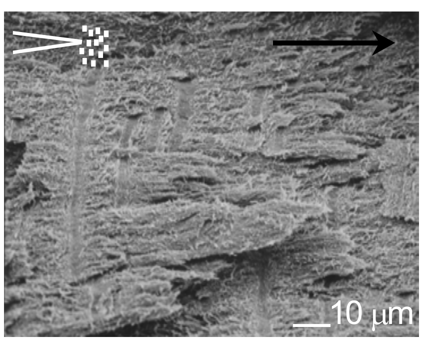
Nalla, Kinney & Ritchie, *Biomater*, in review (2003)

Anisotropy in Toughness in Dentin

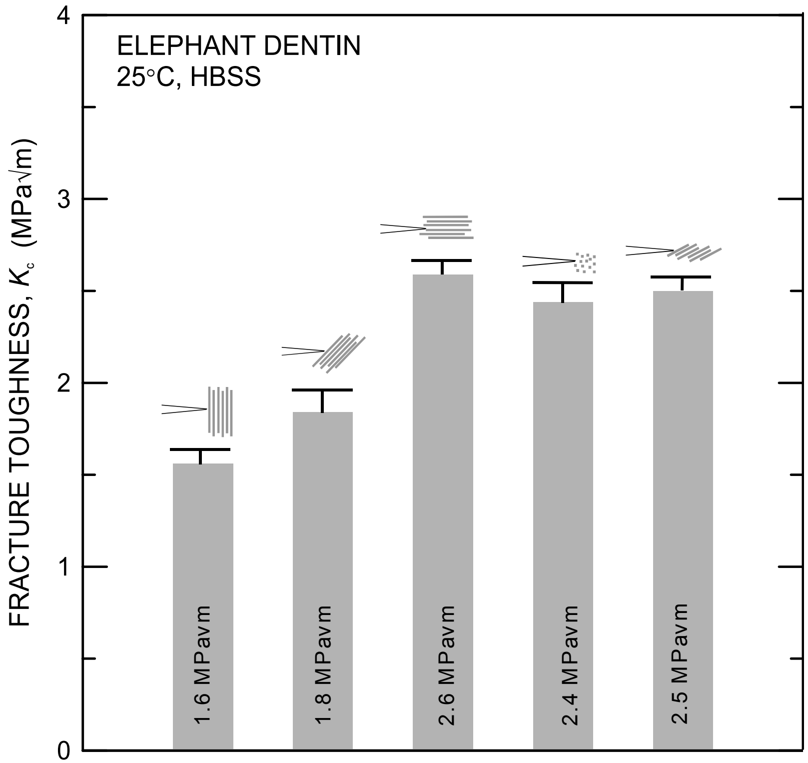


perpendicular orientation

Toughness anisotropic with tubule orientation

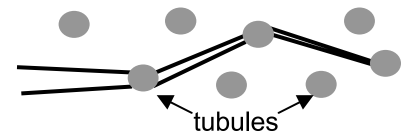


anti-plane parallel orientation

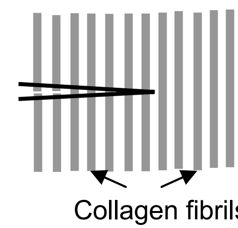


Nalla, Kinney & Ritchie, *Biomater*, in review (2003)

Toughening Mechanisms

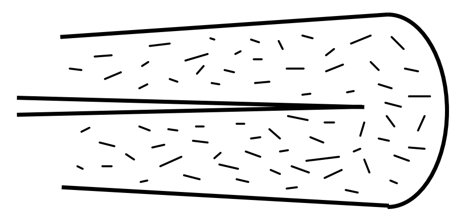


crack deflection



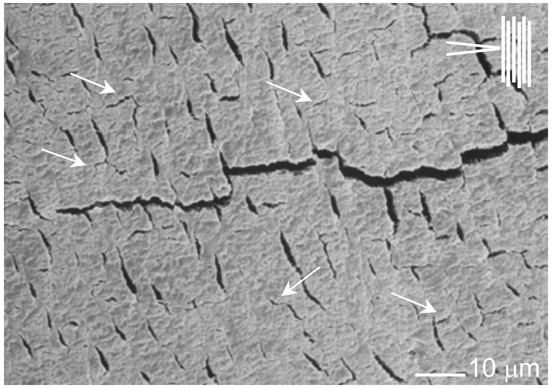
fibril bridging

uncracked ligament bridging



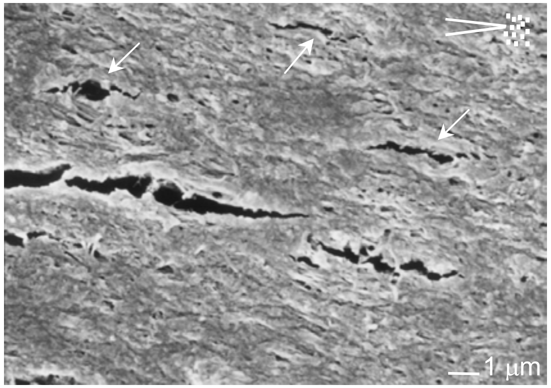
microcracking

Toughening Mechanisms in Dentin



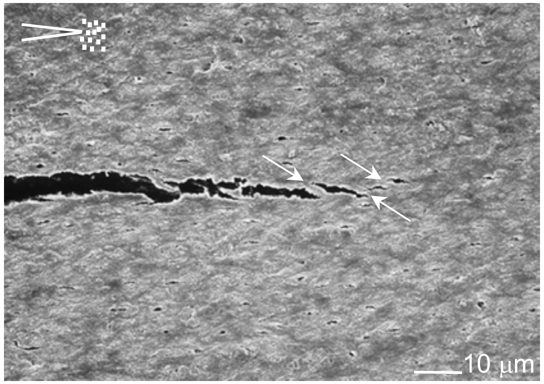
crack growth direction

Crack Deflection
(very localized)



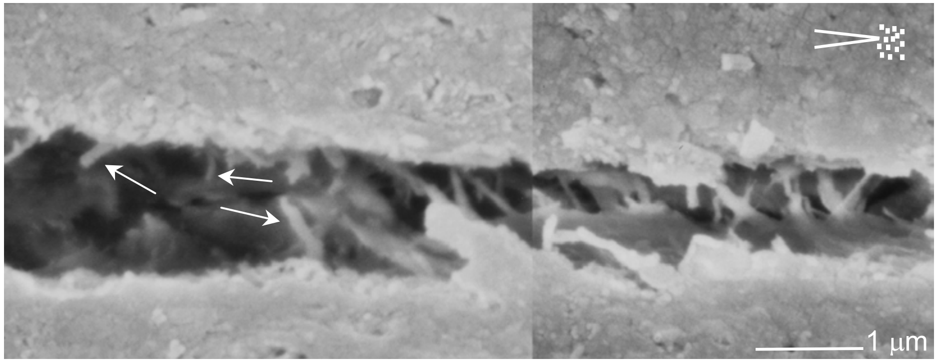
crack growth direction

Microcracking
(K_{mic} ~ 0.3 MPa√m)



crack growth direction

Uncracked Ligament Bridging
(K_b^{ul} ~ 0.1-0.4 MPa√m)

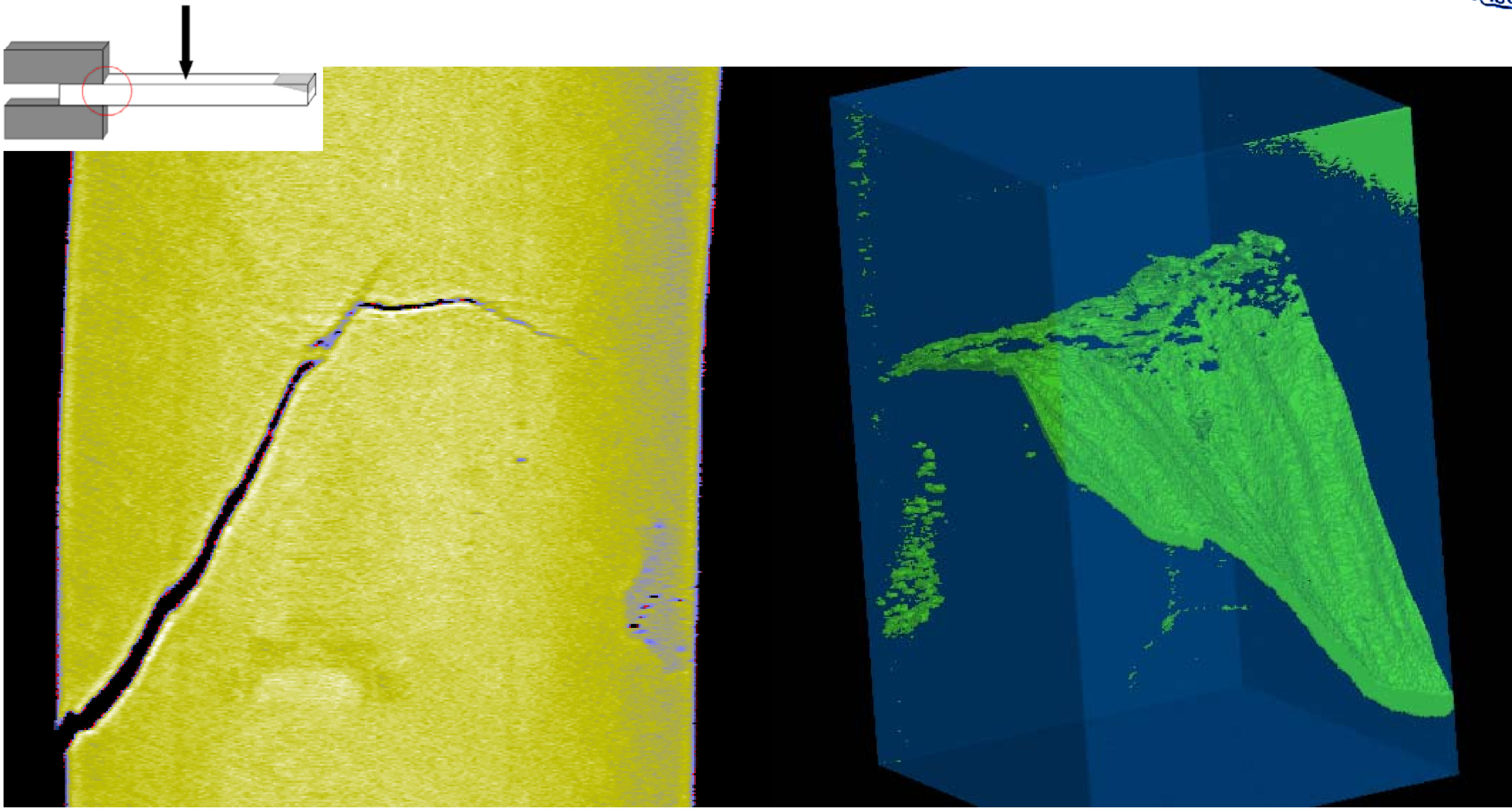


crack growth direction

Collagen Fibril Bridging
(K_b^f < 0.1 MPa√m)

Nalla, Kinney & Ritchie, *Biomater*, in review (2003)

X-Ray Computed Tomography

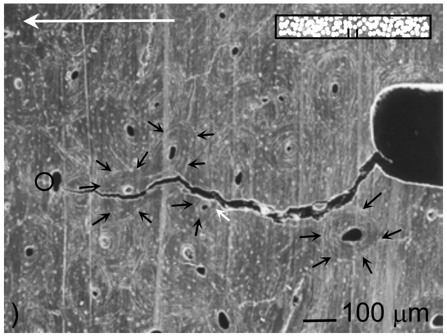


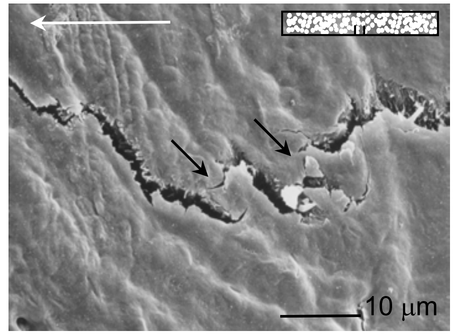
• clear three-dimensional evidence of uncracked ligament bridging

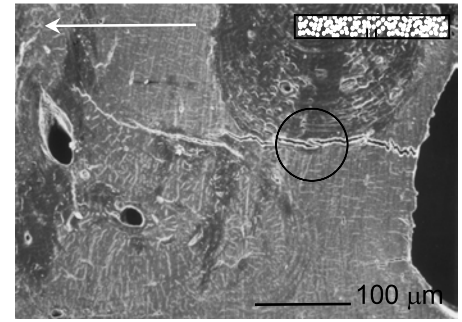
imaged at the Stanford Synchrotron Radiation Laboratory - SSRL, Stanford Linear Accelerator Center - SLAC

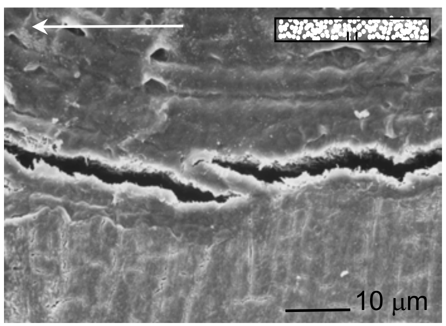
Kinney, Nalla & Ritchie, unpublished research (2002)

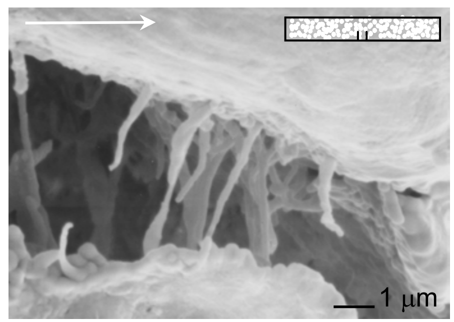
Toughening Mechanisms in Bone

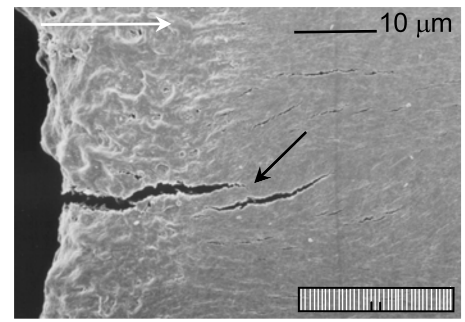


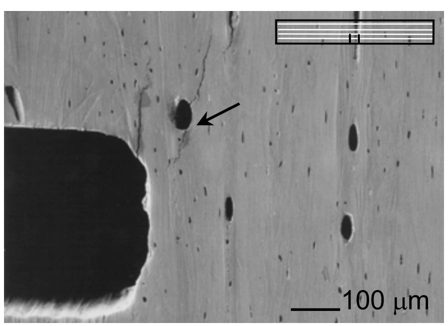












Uncracked Ligament Bridging

$K_b^{ul} \sim 0.3$ MPa$\sqrt{\text{m}}$ (in-plane longitudinal)

Fibril Bridging

$K_b^{f} \sim 0.07$ MPa$\sqrt{\text{m}}$ (anti-plane longitudinal)

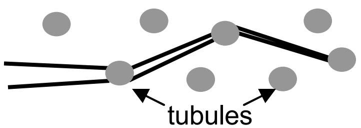
Crack Deflection

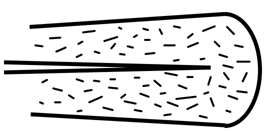
$K_d \sim 2.7$ MPa$\sqrt{\text{m}}$ (transverse)

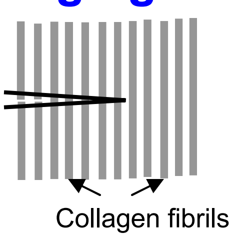
Nalla, Stölken, Kinney & Ritchie, *J Biomech*, in review (2003)

Toughening Mechanisms

Mechanism	Model	Dentin	Bone
Crack Deflection	$k_1(\alpha) = c_{11}(\alpha)\,K_I + c_{12}(\alpha)\,K_{II}$ $k_2(\alpha) = c_{21}(\alpha)\,K_I + c_{22}(\alpha)\,K_{II}$ $K_d = (k_{12} + k_{22})^{1/2}$ (Bilby *et al.*, 1978; Cottrell & Rice, 1980)	very localized (perpendicular)	2.7 MPa√m (transverse)
Microcracking	$K_{mic} = 0.22\,\varepsilon_m E' f_m\, l_m^{1/2} + \beta\, f_m K_c$ (Evans & Fu, 1985: Hutchinson, 1987)	0.3 MPa√m (both)	-
Uncracked Ligament Bridging	$K_b^{ul} = -f_{ul} K_I[(1+l_u/rb)^{1/2}-1]\,/\,[1-f_{ul}+f_{ul}(1+l_{ul}/rb)^{1/2}]$ (Shang & Ritchie, 1989)	0.1-0.4 MPa√m (parallel)	0.3 MPa√m (in-plane long.)
Collagen Fibril Bridging	$K_b^f = 2\,\sigma_b\, f_f\,(2\, l_f\,/\,\pi)^{-1/2}$ (Evans & McMeeking, 1986)	<0.1 MPa√m (parallel)	0.07 MPa√m (anti-plane long.)



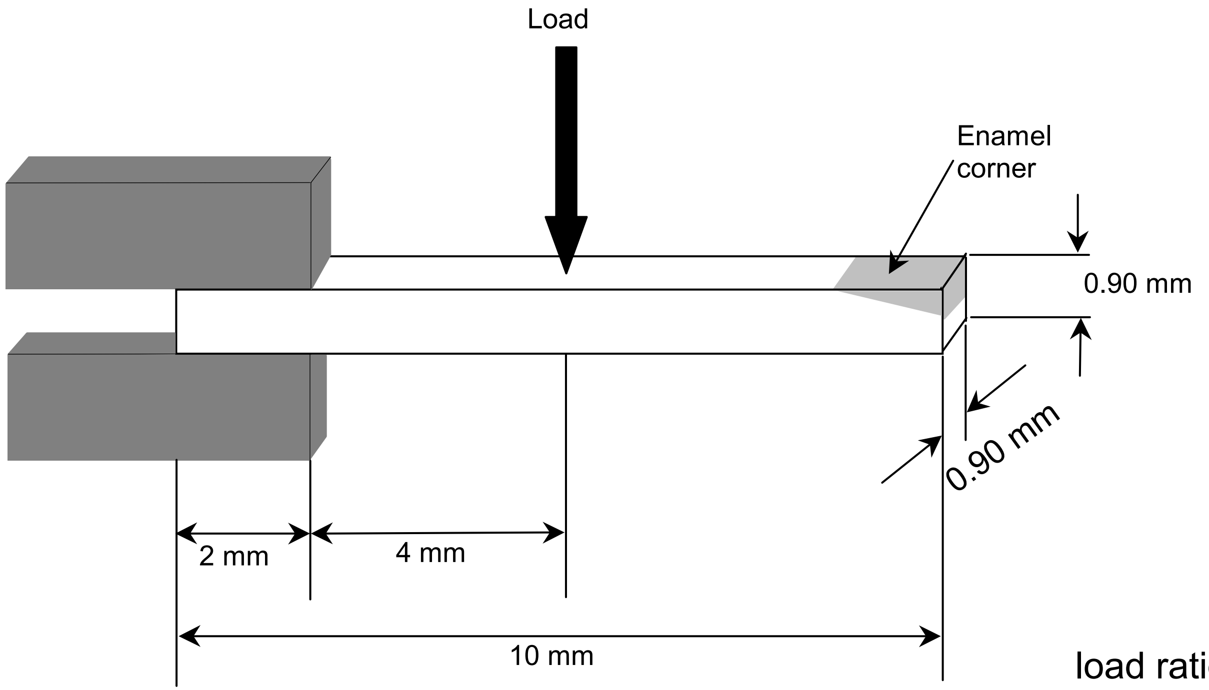




Fatigue of Dentin

- Tonami *et al.*, (1997)
  - 10^5-cycle tensile fatigue strength measured for *bovine* dentin
  - Fatigue strength of 47-51 MPa found: lower value for older animals
  - Tests only over narrow range of purely tensile load ratios, $R \sim 0.15$-0.25
  - tests conducted at 37°C in water; demineralization concerns
  - test duration too short; typical teeth loaded more than 10^6 times annually
- Arola *et al.*, (2002)
  - preliminary crack-growth data for *bovine* dentin
  - zero-tensile ($R \sim 0$) cycling at 25 Hz in a saline bath at 21°C
  - rates of fatigue-crack growth highest perpendicular to the tubules
- Absolutely no data on human dentin

Stress-Life (*S*/*N*) Approach

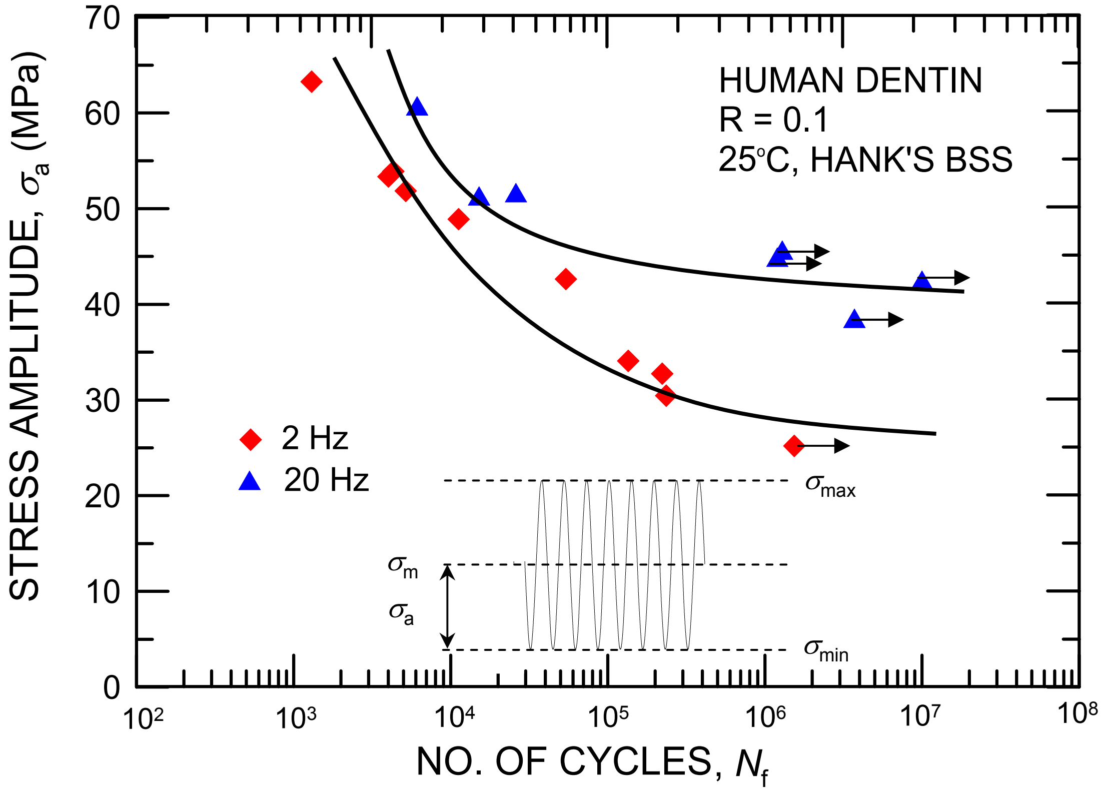


load ratio $R = \sigma_{min}/\sigma_{max}$

- cantilever-beam geometry
- all tests in conducted in Hank's Balanced Salt Solution (HBSS)
- all tests performed on an ELF® 3200 series acoustic testing machine (EnduraTEC Inc., Minnetonka, MN)
- three frequencies, 2 Hz, 10 Hz, 20 Hz; wide range of load ratios, R = -1 to 0.5

Nalla, Imbeni, Kinney, Staninec, Marshall & Ritchie, *J Biomed Mater Res*, in press (2003)

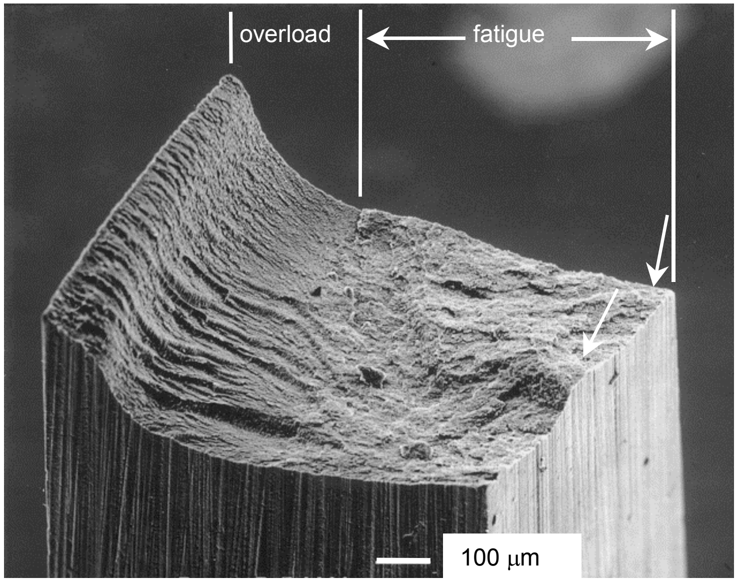
S/*N* Results on Human Dentin



- clear evidence of a susceptibility of human dentin to fatigue
- "metal-like" fatigue *S*/*N* behavior with frequency-dependent fatigue limit at 10^6-10^7 cycles of ~25 and 45 MPa
- fatigue lives, in terms of cycles to failure are shorter at lower frequency

Nalla, Imbeni, Kinney, Staninec, Marshall & Ritchie, *J Biomed Mater Res*, in press (2003)

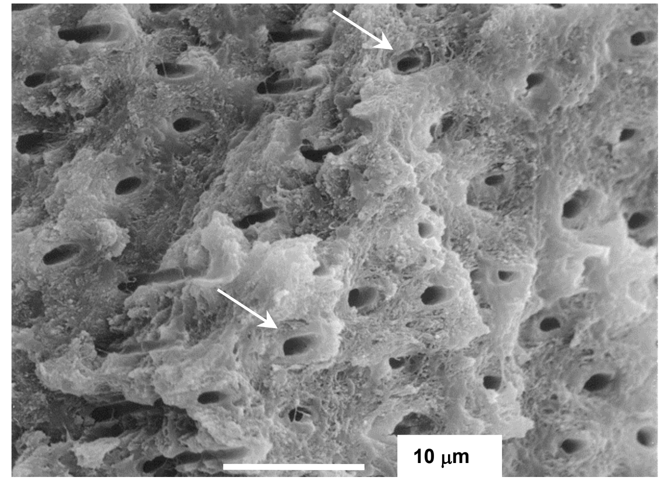
Fractography



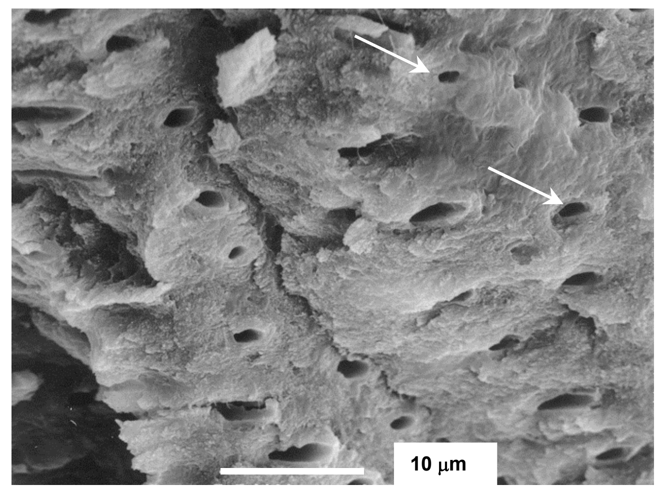
Crack Growth Direction

- Morphology of the fracture surfaces during fatigue-crack propagation essentially identical to overload (catastrophic) failure

Fatigue surface



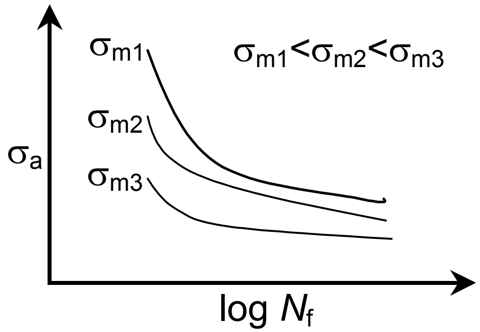
Crack Growth Direction

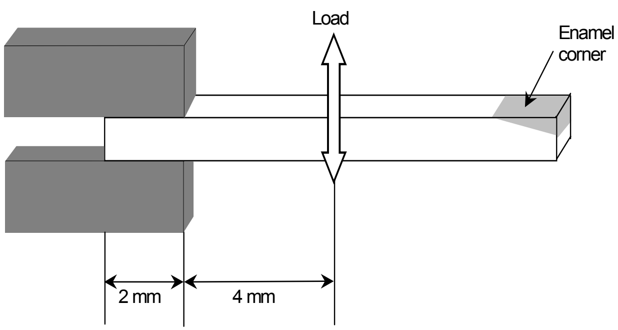


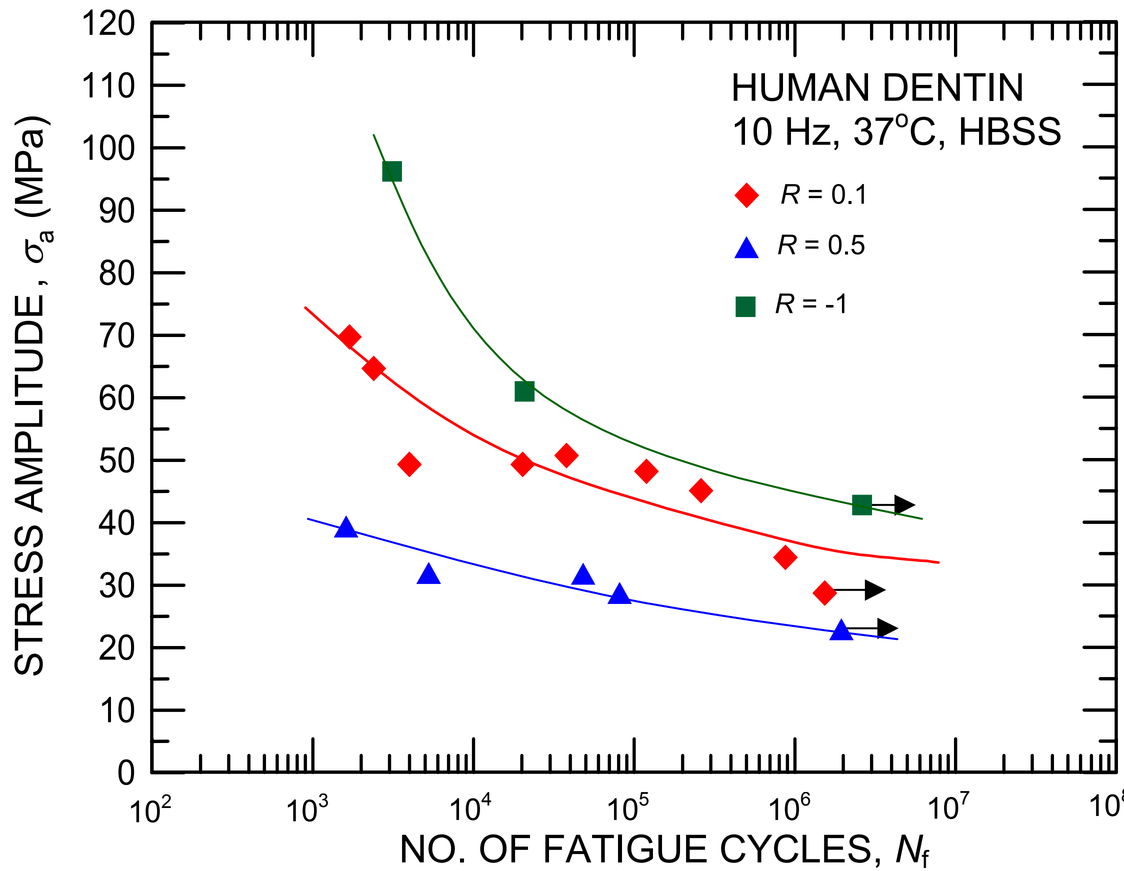
Overload surface

Nalla, Imbeni, Kinney, Staninec, Marshall & Ritchie, *J Biomed Mater Res*, in press (2003)

Effect of Mean Stress (Load Ratio)



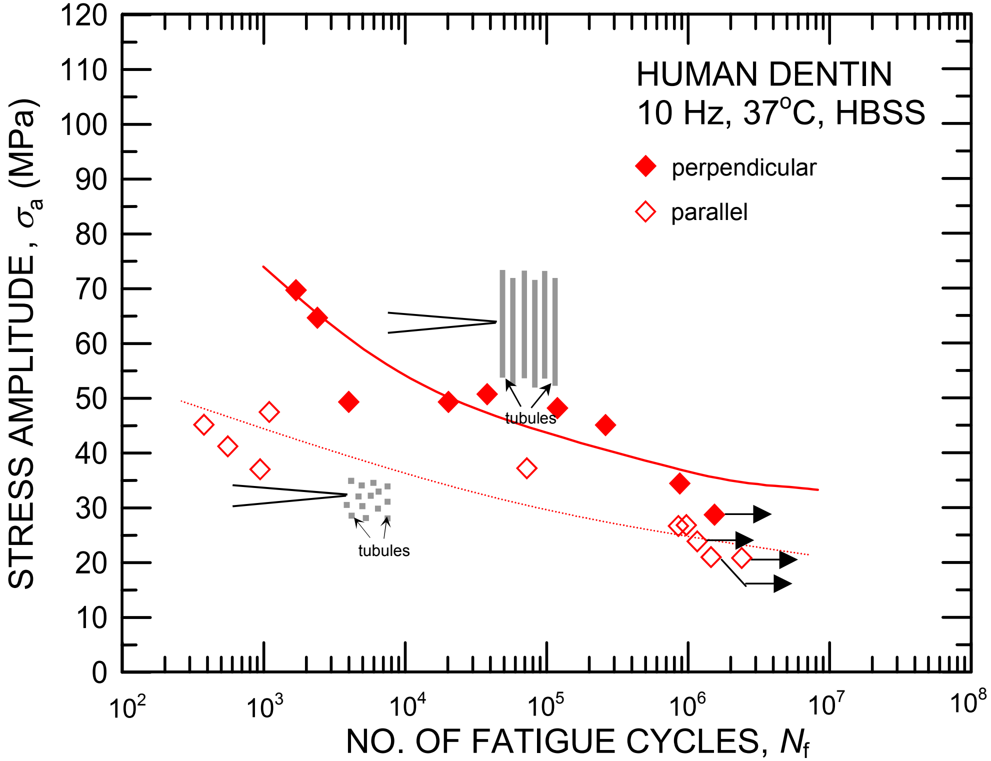


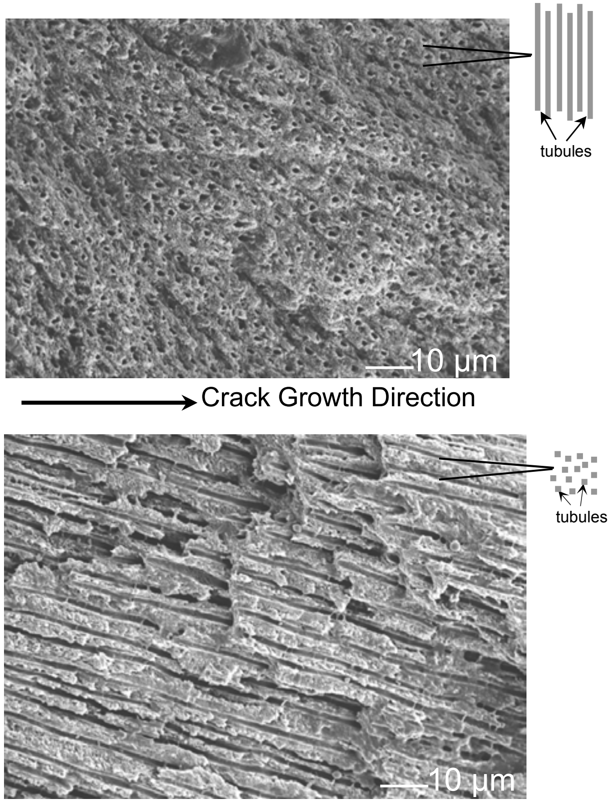


- cantilever bending
- *R* ratios: 0.1, 0.5 and -1
- 10 Hz, 37°C, HBSS

Nalla, Kinney, Marshall & Ritchie, unpublished research (2003)

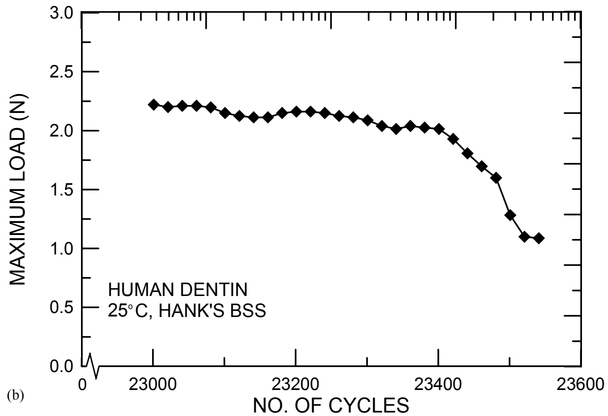
Orientation Effects



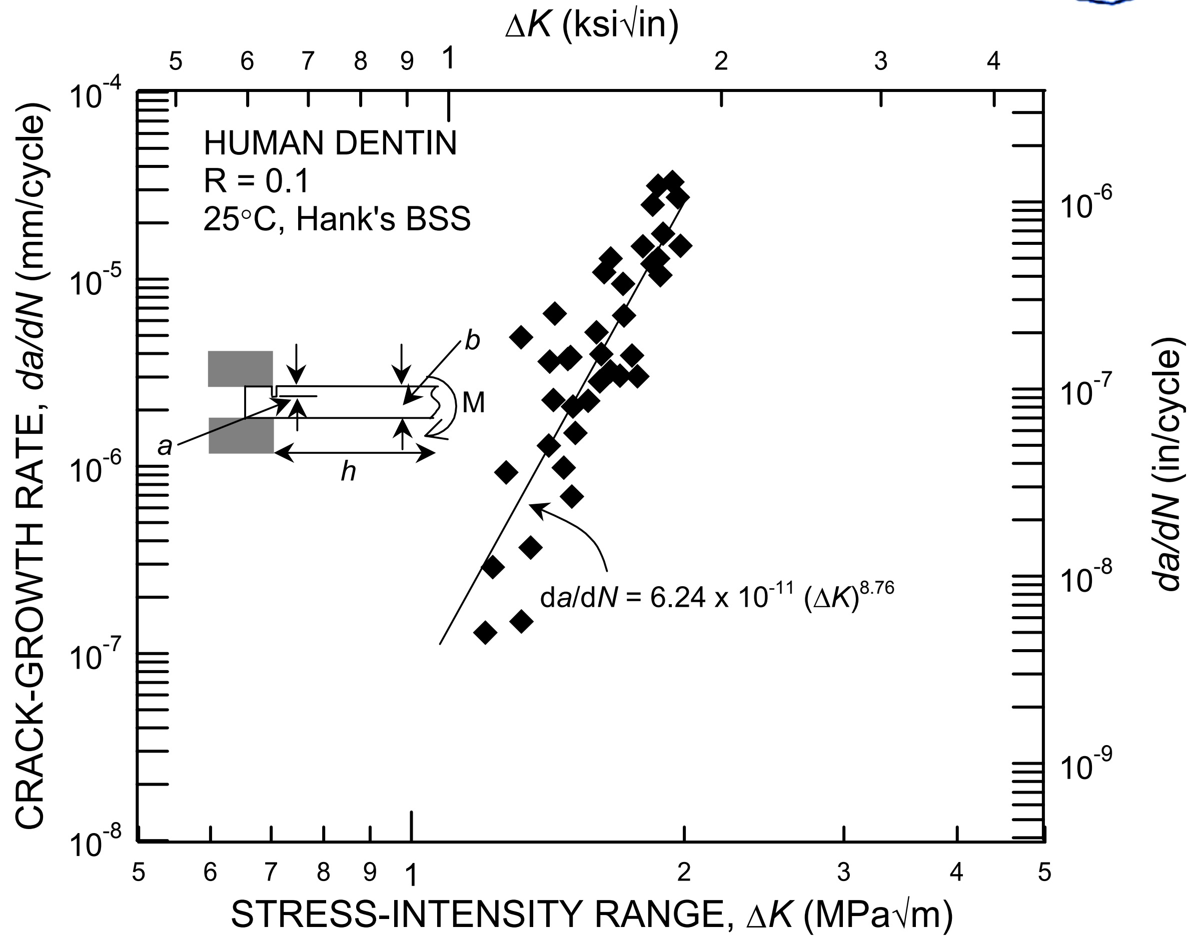


- microstructure affects both LCF and HCF behavior
- lower fatigue limits for *parallel,* as compared to *perpendicular,* orientation
- orientation effect in fatigue contrary to that seen for toughness

Fatigue-Crack Growth in Human Dentin



- decay in stiffness used to estimate crack lengths



- Paris power-law relationship, $da/dN = C\,\Delta K^m$, where exponent $m \sim 8.76$
- Estimated fatigue threshold, $\Delta K_{TH} \sim 1.06$ MPa√m, ~60% of the fracture toughness

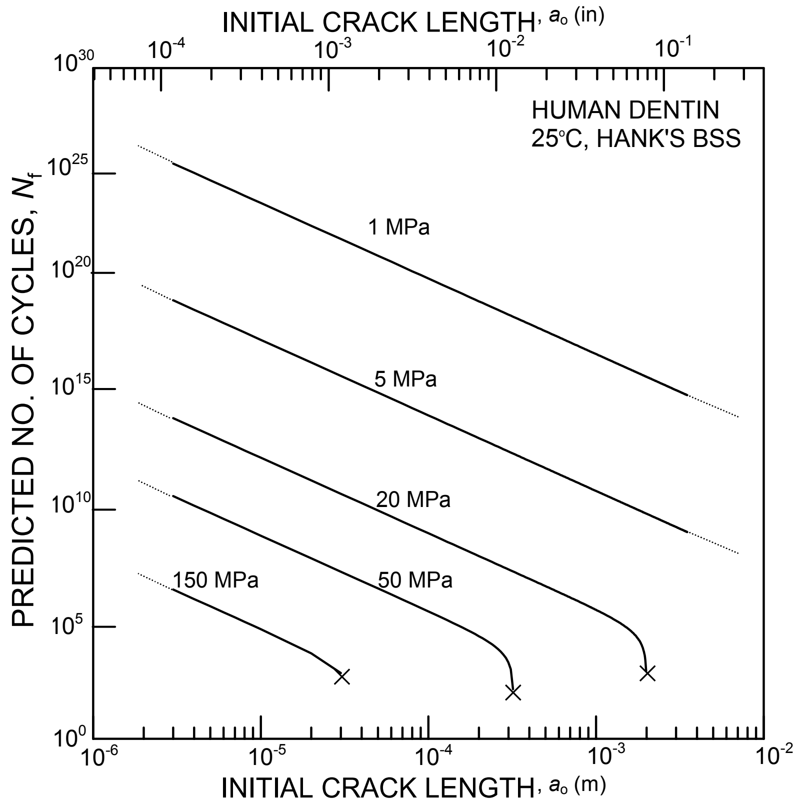
Nalla, Imbeni, Kinney, Staninec, Marshall & Ritchie, *J Biomed Mater Res*, in press (2003)

Damage-Tolerant Lifetime Prediction

- Integrating the Paris equation, from an initial, a_o, to final, a_c, crack size:

$$N_f = 2\,(f(a/b)\Delta\sigma_{app})^{-m}\,(m-2)^{-1}\,C^{-1}\,\pi^{-m/2}\,[a_o^{1-m/2} - a_c^{1-m/2}]$$

- for initial flaw size, $a_o \sim 100\ \mu m$, projected life at $\sigma_{app} \sim 20$ MPa over a billion cycles
- for a 600 μm flaw, projected life drops to ~3.6 million cycles, or 3 to 4 years
- for a 900 μm flaw, projected life as low as a few months
- for *in vivo* stresses of 5-20 MPa, small flaws in teeth, ~ 250 μm, will not radically affect their structural integrity, as predicted fatigue lifetimes exceed patient lifetimes



Nalla, Imbeni, Kinney, Staninec, Marshall & Ritchie, *J Biomed Mater Res*, in press (2003)

Summary

- First accurate fracture toughness of human dentin measured.
- Critical fracture event in dentin consistent with a *strain-based* criterion.
- Effect of orientation on toughness defined; lowest toughness measured for fracture perpendicular to dentinal tubules.
- Toughness in dentin arises from extrinsic toughening mechanisms: collagen fibril and uncracked ligament bridging, microcracking, crack deflection.
- Human dentin shown to be susceptible to fatigue failure.
- “Metal-like” *S/N* behavior seen, sensitive to both frequency and mean stress, with a 10^6-10^7 cycle fatigue limit of 25-45 MPa.
- Rudimentary life-prediction analyses indicates that flaws of up to 250 μm in size will not radically affect structural integrity under typical physiological loads.